



# UNITED STATES PATENT AND TRADEMARK OFFICE

UNITED STATES DEPARTMENT OF COMMERCE  
United States Patent and Trademark Office  
Address: COMMISSIONER FOR PATENTS  
P.O. Box 1450  
Alexandria, Virginia 22313-1450  
www.uspto.gov

APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
10/584,301	08/07/2007	Henrik Jensen	55320.000402	9824

21967 7590 02/03/2010  
HUNTON & WILLIAMS LLP  
INTELLECTUAL PROPERTY DEPARTMENT  
1900 K STREET, N.W.  
SUITE 1200  
WASHINGTON, DC 20006-1109

EXAMINER
----------

HAN, SHENG

ART UNIT	PAPER NUMBER
----------	--------------

1793

MAIL DATE	DELIVERY MODE
-----------	---------------

02/03/2010

PAPER

**Please find below and/or attached an Office communication concerning this application or proceeding.**

The time period for reply, if any, is set in the attached communication.

<b>Office Action Summary</b>	<b>Application No.</b> 10/584,301	<b>Applicant(s)</b> JENSEN ET AL.	
	<b>Examiner</b> SHENG HAN	<b>Art Unit</b> 1793	

**-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --**

### Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

### Status

- 1) ☐ Responsive to communication(s) filed on \_\_\_\_.
- 2a) ☐ This action is **FINAL**.                      2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

### Disposition of Claims

- 4) ☒ Claim(s) See Continuation Sheet is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 1, 6, 7, 9, 11, 13, 15, 17, 18, 25, 30, 32, 36, 39, 43, 51, 52, 92, 93, 97, 98, 112, 114 and 116 is/are rejected.
- 7) ☒ Claim(s) \_\_\_\_ is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_ are subject to restriction and/or election requirement.

### Application Papers

- 9) ☐ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on \_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.  
Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).  
Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

### Priority under 35 U.S.C. § 119

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All    b) ☐ Some \*    c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
  2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_.
  3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

### Attachment(s)

- |  |   |
|--|---|
| 1) <input type="checkbox"/> Notice of References Cited (PTO-892)                       | 4) <input type="checkbox"/> Interview Summary (PTO-413)           |
| 2) <input type="checkbox"/> Notice of Draftperson's Patent Drawing Review (PTO-948)    | Paper No(s)/Mail Date. ____.                                      |
| 3) <input checked="" type="checkbox"/> Information Disclosure Statement(s) (PTO/SB/08) | 5) <input type="checkbox"/> Notice of Informal Patent Application |
| Paper No(s)/Mail Date ____.  | 6) <input type="checkbox"/> Other: ____.                          |

Continuation of Disposition of Claims: Claims pending in the application are  
1,6,7,9,11,13,15,17,18,25,30,32,36,39,43,51,52,92,93,97,98,112,114 and 116.

## **DETAILED ACTION**

### ***Response to Arguments***

As to Applicant's arguments filed 11/13/09 on the Reverchon reference, they have been fully considered but they are not persuasive over this reference.

Specifically, Applicant contends that Reverchon does not teach the use of a solid reactor filling material. On the contrary, Reverchon teaches use a stainless steel basket filled with glass Raschig rings. This was not made clear in the previous rejection and is now more clearly stated herein. Glass and stainless steel baskets can come in many forms, including fibrous ones. This is well known in the art (see 2004/0245496, para. 0069, glass fibers, 2004/0091604, para. 0051, use of glass fibers, 2005/0214533, steel fibers used as substrate, 5977004, col. 7, line 66 steel and stainless steel fibers). This is used partly to overcome the problem previously found in these reactors of catalytic reactions occurring on the reactor walls of the system (pg. 253, para. 2 of Reverchon).

As to the Merzbacher reference, Tillotson, Applicant's arguments, have been fully considered and are persuasive. Therefore, the rejection has been withdrawn. However, upon further consideration, a new ground(s) of rejection is made (see below). As to the fact that both Merzbacher and Tillotson use the supercritical CO<sub>2</sub> after the reaction, these references are no longer used based on this step (see rejection below).

As to the Lauf reference, Applicant's arguments, have been fully considered and are persuasive. Therefore, the rejection has been withdrawn.

***Claim Rejections - 35 USC § 112***

1. The following is a quotation of the second paragraph of 35 U.S.C. 112:

The specification shall conclude with one or more claims particularly pointing out and distinctly claiming the subject matter which the applicant regards as his invention.

Claims 13, 15, 17, 18, 25, 30, 32, 36, 39, 43, 51, 52, 92, 93, 97, 98 are rejected under 35 U.S.C. 112, second paragraph, as being indefinite for failing to particularly point out and distinctly claim the subject matter which applicant regards as the invention.

Regarding Claims 13, 15, 17, 18, 25, 30, 32, 36, 39, 43, 51, 52, 92, 93, 97, 98 these claims partly depends on cancelled claims.

***Claim Rejections - 35 USC § 102***

The following is a quotation of the appropriate paragraphs of 35 U.S.C. 102 that form the basis for the rejections under this section made in this Office action:

A person shall be entitled to a patent unless –

(a) the invention was known or used by others in this country, or patented or described in a printed publication in this or a foreign country, before the invention thereof by the applicant for a patent.

Claims 1, 15, 92 and 116 are rejected under 35 U.S.C. 102(a) as being anticipated by Reverchon et al. ("Synthesis of Titanium Hydroxide nanoparticles in Supercritical Carbon Dioxide on the Pilot Scale").

As to Claims 1, 15 and 92, Reverchon discloses several methods of producing titanium hydroxide (Ti(OH)<sub>4</sub>) and titanium oxide (TiO<sub>2</sub>) by using known sol-gel preparation methods and modifications thereof (introduction). One of these methods

Art Unit: 1793

discussed by Reverchon is taught by Tadros, which uses a hydrolysis method of a titanium-precursor (titanium tetra-isopropoxide (TTIP)) by mixing TTIP with water, a surfactant and supercritical carbon dioxide (SC-CO<sub>2</sub>) to form Ti(OH)<sub>4</sub> microparticles with a particle size distribution of between 0.1 to 2.0 microns (pg. 254, col. 1, para. 2). Another similar process described by Reverchon uses the same compounds in different order and at different pressure and temperatures (30 MPa at 50 degrees Celsius, pg. 254, col. 1, para. 2). In general, Reverchon describes the process as following the general order of the following formula:



In the specific invention described by Reverchon, the author teaches the formation of Ti(OH)<sub>4</sub> using various starting materials including CO<sub>2</sub>, CO<sub>2</sub>-TTIP and CO<sub>2</sub>-H<sub>2</sub>O and a liquid separator (pg. 254, sec. 2). Here, TTIP serves as the semi-metal containing precursor and water is the co-solvent (pg. 254, sec. 2, para. 3). A supercritical solvent comprising carbon dioxide is used in the reaction (pg. 254, section 2, para. 3, SC-CO<sub>2</sub>). CO<sub>2</sub>-H<sub>2</sub>O is also used, and can function as either the reactant, or the solid reactor filling material, or both (pg. 254, section 2, para. 3). Finally, a liquid separator (pg. 254, section 2, para. 1, "LS") and silica gel (pg. 254, section 2, para. 4) are both added to this reaction and can function as one of the reaction starting materials. Since TTIP (the semi-metal) is mixed with H<sub>2</sub>O, they are inherently in contact with each other. Reverchon teaches use a stainless steel basket filled with glass Raschig rings in the reaction chamber. Finally, the process results in the formation of Ti(OH)<sub>4</sub> (page 254, section 2, para. 1).

Regarding Claim 7, Reverchon teaches that the product is amorphous (pg. 254, col.1 or pg. 259, col. 2, para. 1).

Regarding Claim 9, Reverchon teaches that the product is amorphous (pg. 254, col.1 or pg. 259, col. 2, para. 1).

Regarding Claim 11, Reverchon teaches that the phases of the material can be both amorphous and crystalline (pg. 259, col. 2, para. 1, amorphous state more than crystals, but both are present).

Regarding Claim 17, Reverchon teaches use of water.

Regarding Claim 25, Reverchon does not teach a temperature above 400 degrees C. Since the other temperature ranges are preferable, they are not required limits in this claim.

Regarding Claim 30, Reverchon teaches use of 30 Mpa, which is about 300 bar at a temperature of about 50 degrees C (pg. 254, col. 1). Since the other temperature ranges are alternatives and not required limitations they are not addressed herein. Also the supercritical solvent is CO<sub>2</sub>.

Art Unit: 1793

Regarding Claim 36, Reverchon teaches that the solutions were mixed for about 15 seconds and then gelled for 1 minute (col. 6, lines 35-41).

Regarding Claim 43, Reverchon teaches use of a titanium alkoxide (pg. 253, para. 2, TTIP).

Regarding Claim 51, Reverchon teaches use of water as the solvent.

Regarding Claim 116, Reverchon teaches that the compound can be mixed with anhydrous methyl alcohol with water ammonium hydroxide and water (col. 6, lines 30-31).

***Claim Rejections - 35 USC § 103***

2. The text of those sections of Title 35, U.S. Code not included in this action can be found in a prior Office action.

Claim 6, 13, 32 and 112 are rejected under 35 U.S.C. 103(a) as being unpatentable over Reverchon.

Regarding Claim 6, Reverchon teaches that this process is known to be made by the Sol-gel method (pg. 253, para. 1).



Regarding Claim 13, Reverchon teaches all the features described in the rejection of Claim 1. Reverchon does not teach any particular order. It would have been obvious to one of ordinary skill in the art at the time of the invention that the additives of this invention could be applied in any order capable of producing the same product however by optimization and experimentation.

Regarding Claim 32, Reverchon teaches operating the process as described in the rejection to Claim 1 at a pressure of 10MPa (Introduction) and a temperature of between 258 to 295 degrees Celsius (Introduction). Reverchon also teaches a higher pressure (Pg. 254, col. 1, para. 2, 30MPa), a lower temperature (pg. 254, col. 1, para. 2, 50 degrees Celsius). It would have been obvious to one of ordinary skill in the art at the time of the invention to optimize the pressure and temperature of the reaction according to the desired product. As for the supercritical isopropanol, it would have been obvious to one of ordinary skill in the art at the time of the invention to use any known type of alcohol as a supercritical solvent, such as isopropanol as taught by Reverchon, because it is known to be useable in formation of the desired titania product.

Regarding Claim 112, Reverchon discloses several methods of producing titanium hydroxide ( $\text{Ti}(\text{OH})_4$ ) and titanium oxide ( $\text{TiO}_2$ ) by using known sol-gel preparation methods and modifications thereof (introduction). One of these methods discussed by Reverchon is taught by Tadros, which uses a hydrolysis method of a titanium-precursor (titanium tetra-isopropoxide (TTIP)) by mixing TTIP with water, a

Art Unit: 1793

surfactant and supercritical carbon dioxide (SC-CO<sub>2</sub>) to form Ti(OH)<sub>4</sub> microparticles with a particle size distribution of between 0.1 to 2.0 microns (pg. 254, col. 1, para. 2). Another similar process described by Reverchon uses the same compounds in different order and at different pressure and temperatures (30 MPa at 50 degrees Celsius, pg. 254, col. 1, para. 2). In general, Reverchon describes the process as following the general order of the following formula:



In the specific invention described by Reverchon, the author teaches the formation of Ti(OH)<sub>4</sub> using various starting materials including CO<sub>2</sub>, CO<sub>2</sub>-TTIP and CO<sub>2</sub>-H<sub>2</sub>O and a liquid separator (pg. 254, sec. 2). Here, TTIP serves as the semi-metal containing precursor and water is the co-solvent (pg. 254, sec. 2, para. 3). A supercritical solvent comprising carbon dioxide is used in the reaction (pg. 254, section 2, para. 3, SC-CO<sub>2</sub>). CO<sub>2</sub>-H<sub>2</sub>O is also used, and can function as either the reactant, or the solid reactor filling material, or both (pg. 254, section 2, para. 3). Finally, a liquid separator (pg. 254, section 2, para. 1, "LS") and silica gel (pg. 254, section 2, para. 4) are both added to this reaction and can function as one of the reaction starting materials. Since TTIP (the semi-metal) is mixed with H<sub>2</sub>O, they are inherently in contact with each other. Reverchon teaches use a stainless steel basket filled with glass Raschig rings in the reaction chamber. The particle size of the titania can be a microparticle (pg. 254, col. 1). Finally, the process results in the formation of Ti(OH)<sub>4</sub> (page 254, section 2, para. 1). Although Reverchon does not specifically teach applying this process to an apparatus, it would have been obvious to one of ordinary skill in the

Art Unit: 1793

art at the time of the invention that a means for providing each of these ingredients would be used.

Claims 1, 6, 18, 92, 112 and 114 are rejected under 35 U.S.C. 103(a) as being unpatentable over Merzacher (6296678) and further in view of Reverchon.

As to Claims 1 and 92, Merzbacher discusses a method of making metal powders (col. 1, lines 21-25) that are ultra-fine in size (col. 3, lines 1-2) using a modified sol-gel reaction (col. 2, lines 3-5). In one example disclosed by Merzbacher, a silica compound was prepared by mixing a silica-based precursor (tetramethoxysilane, col. 6, lines 29-30) with anhydrous methyl alcohol (col. 6, line 30), water ammonium hydroxide (col. 6, line 31) and water (col. 6, line 31). Since all these compounds were mixed together, the semi-metal and the co-solvent come into contact with each other. Other components were used in the synthesis process such as a phosphorus powder (col. 6, line 38) or a gel (col. 6, line 41). Supercritical CO<sub>2</sub> was used to dry the composition (col. 6, lines 44-45).

Although Merzbacher teaches use of the supercritical CO<sub>2</sub> for drying the compound, it does not teach use of it concurrent to the reaction.

Reverchon discloses several methods of producing titanium hydroxide (Ti(OH)<sub>4</sub>) and titanium oxide (TiO<sub>2</sub>) by using known sol-gel preparation methods and modifications thereof (introduction). One of these methods discussed by Reverchon is taught by Merzbacher, which uses a hydrolysis method of a titanium-precursor (titanium

Art Unit: 1793

tetra-isopropoxide (TTIP)) by mixing TTIP with water, a surfactant and supercritical carbon dioxide (SC-CO<sub>2</sub>) to form Ti(OH)<sub>4</sub> microparticles with a particle size distribution of between 0.1 to 2.0 microns (pg. 254, col. 1, para. 2). Another similar process described by Reverchon uses the same compounds in different order and at different pressure and temperatures (30 MPa at 50 degrees Celsius, pg. 254, col. 1, para. 2). In general, Reverchon describes the process as following the general order of the following formula:



In the specific invention described by Reverchon, the author teaches the formation of Ti(OH)<sub>4</sub> using various starting materials including CO<sub>2</sub>, CO<sub>2</sub>-TTIP and CO<sub>2</sub>-H<sub>2</sub>O and a liquid separator (pg. 254, sec. 2). Here, TTIP serves as the semi-metal containing precursor and water is the co-solvent (pg. 254, sec. 2, para. 3). A supercritical solvent comprising carbon dioxide is used in the reaction (pg. 254, section 2, para. 3, SC-CO<sub>2</sub>). CO<sub>2</sub>-H<sub>2</sub>O is also used, and can function as either the reactant, or the solid reactor filling material, or both (pg. 254, section 2, para. 3). Finally, a liquid separator (pg. 254, section 2, para. 1, "LS") and silica gel (pg. 254, section 2, para. 4) are both added to this reaction and can function as one of the reaction starting materials. Since TTIP (the semi-metal) is mixed with H<sub>2</sub>O, they are inherently in contact with each other. Reverchon teaches use a stainless steel basket filled with glass Raschig rings in the reaction chamber. Finally, the process results in the formation of Ti(OH)<sub>4</sub> (page 254, section 2, para. 1).

It would have been obvious to one of ordinary skill in the art at the time of the invention to apply the supercritical CO<sub>2</sub> step to the formation of titania particles during the reaction rather than after, as taught by Reverchon, with the process as described by Merzacher because it is known in the art, as explained by Reverchon that not only is supercritical CO<sub>2</sub> useable for drying (pg. 253, para. 2), but facilitates thermal decomposition of the titania precursor into the aqueous solution.

Regarding Claim 6, Merzacher teaches that the process is made by the sol-gel process (col. 2, line 6).

Regarding Claim 18, Merzacher teaches that the substitution source can be a carbon based material (col. 3, lines 11-12 or col. 5, lines 49-50, resorcinol formaldehyde aerogel).

Regarding Claims 112 and 114, Merzacher teaches that the process described above is done with an apparatus shown in Figure 1. It is noted that the rejection is over apparatus claims not method claims. The prior art only has to provide a structure that is capable of performing in the manner claimed and not necessarily have ever been intended to be used in this manner. As such, apparatus claims 112 and 114 requires a reaction chamber that can hold a composition; at least one inflow channels that

Art Unit: 1793

communicate with the chamber, that channel being capable of supplying a compound listed in Claims 112 or 114. It is the examiner's position that Merzacher meets the limitations of the instant claims. If the applicant were to establish that significant structural differences exist with the prior art apparatus which would make it incapable of performing the evacuation step and amend the claims appropriately, the art rejection over the apparatus claims will be withdrawn and the case pass to issue.

Claims 39, 52, 92, 93, 97 and 98 are rejected under 35 U.S.C. 103(a) as being unpatentable over Reverchron or Merzacher in view of Reverchron as applied to claim 1 above, and further in view of Tillotson.

Reverchron or Merzacher in view of Reverchron teach a method of making a titania compound using a titania-precursor, water, a supercritical solvent and other possible components such as glass Raschig rings or stainless steel basket compounds inside, but do not specifically teach use of different metal or semi metal containing precursors along with the primary precursor.

Tillotson instructs on a method of making a metal oxide material using a modified sol-gel process (abstract and para. 0026). In one process, Tillotson teaches using a metal salt reduction process route (para. 0032), a metal alkoxide precursor route (para. 0034), or the process whereby other solvents are used with water (para. 0037).

Art Unit: 1793

According to Tillotson, one method of making the metal oxide is to dissolve a metal salt or metal alkoxide in a solvent to form a metal salt solution or a metal alkoxide solution (claim 1). Tillotson teaches that one can optionally add a proton scavenger to the solution (claim 1), which can be a hydrocarbon-based compound or an alcohol (para. 0032).

Afterwards, a fuel source is added to the mixture (Claim 1), such as a particulate metal (para. 0058). This composition can also comprise polymers (para. 0063). Since the metal salt or metal alkoxide is in a solvent, it is therefore also in contact with the solvent. This mixture is then followed by a supercritical extraction (claim 5) wherein the compound can be carbon dioxide (para. 0020). Furthermore, this reaction is made in a series of beakers which can be construed as a reactor. Additionally, it would have been obvious to one of ordinary skill in the art at the time of the invention to use a reactor in order to vary the temperature and pressure of the composition.

Tillston teaches that the fuel added to the mixture can be a particulate metal (para. 0058), which is an additional metal added to the metal alkoxide.

It would have been obvious to one of ordinary skill in the art at the time of the invention that other metal-based compounds, such as that described by Tilliston could be used in the process described by Reverchron, or Merzacher and Reverchron because it is known that other compounds can be mixed into the solution to create the same product.

Regarding Claim 52, Tillotson explains that other solvents may be used in addition to water (para. 0037).

Regarding Claim 92, Tillotson explains that a long list of possible compounds can be used in the mixture, including trimethylene oxide (para. 0032).

Regarding Claim 93, Tillotson describes use of an oxide material (para. 0032) that serves as the matrix for the seed formation. This matrix can be construed as the collecting agent.

Regarding Claim 97, Tillotson teaches formation of a metal-oxide oxidizer skeletal structure whereby fuel is added to the matrix (para. 0068). Since this solid matrix forms, it would have been obvious to one of ordinary skill in the art at the time of the invention that it would be separable from the crystallites formed within the spaces in the structure and reusable as filling material.

Regarding Claim 98, Tillotson explains that the compound can be separable using a super-critical extraction or non-supercritical extraction of the liquid phase, such as with low temperature evaporation (para. 0068).



***Conclusion***

Any inquiry concerning this communication or earlier communications from the examiner should be directed to SHENG HAN whose telephone number is (571)270-5823. The examiner can normally be reached on Monday-Thursday, 8:00-5:30pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Melvin Curtis Mayes can be reached on 571-272-1234. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

Sheng Han  
Examiner  
Art Unit 1793

January 25, 2010

/Melvin Curtis Mayes/  
Supervisory Patent Examiner, Art Unit 1793